Ammonia

OBJECTIVE

Ammonia is a natural component of marine sediments and common constituent of municipal effluents. Ammonia may occur in concentrations that are toxic to marine organisms in toxicity tests; therefore, it must be accurately measured in order to rule out interference with toxicity test results.

EQUIPMENT

Hach DR/2010 spectrophotometer
Test-n-Tube kits (High- or Low-Range)
water quality vials/rack
test tube rack
Nanopure water
0.1 M Ammonium Chloride Standard
0.1 mg/L Ammonium Chloride Standard
scissors
safety equipment: Nitrile gloves, lab coat, lab glasses

STANDARD PREPARATION

If performing a High-Range test, prepare a 10 mg/L NH_3 standard by adding 588 µL of 0.1M NH_3-N ammonium chloride standard to a 100- mL volumetric flask and filling with distilled water. Mix well. If performing a Low-Range test, use the 1.0 mg/L NH_3-N standard.

TEST PREPARATION

- Select the appropriate Hach Test-n-Tube (TNT) test range for the bioassay.
- Label enough TNT test tubes for each sample, and a blank and standard for each range tested.
- Turn on spectrophotometer (lid closed), and allow it to self-test.
- Enter test number (type 343 for HR, or 342 for LR, then ENTER).
- Dial in wavelength if instructed by the machine to do so.
- Aliquot blank or dilution water (distilled) as necessary.
- Count out the number of reagent powder pillows needed for the entire set of samples (one of each for each TNT tube).

TEST INITIATION

- Tap salicylate powder pillow and open. Empty contents into the blank tube.
- Tap cyanurate powder pillow and open. Empty contents into the blank tube.
- Cap tube tightly and shake well. Wipe tube with a tissue.

- Start timer on spectrophotometer (SHIFT-TIMER).
- Aliquot the first sample (100 µl for HR, 2 ml for LR) into TNT tube, and repeat first three steps.
- Aliquot remaining samples, repeating first three steps after each. Remember to change sample volumes when diluting samples or changing test range.
- Continue to shake samples until all solids dissolve.

AMMONIA MEASUREMENT

- When timer goes off (after 20 minutes), place COD tube holder in machine with line facing to your right.
- Blank the machine (place blank vial in COD holder and press ZERO).
- Toggle with arrow key to read NH_3.
- Read blank ammonia concentration and record.
- Wait approximately 1 minute before reading each sample. Record sample and standard ammonia concentrations on the appropriate data sheet.
- NOTE: when reading 1.0 or 10.0 mg/L NH_3-N standard, toggle display to read N.
- If sample is out of range, machine will display UR (under-range) or OR (over-range). If UR, record UR. If OR, dilute sample in half and re-measure.

QUALITY CONTROL

Read each blank and standard, and record on the appropriate data sheet. Blank should read 0.0-0.1 mg/L NH_3. If blank measurements deviate from this value, notify supervisor. Standards should read 1.0 mg/L N or 10.0 mg/L NH_3, as appropriate, or within 10% of these values. If standard measurements deviate by greater than 10%, all measured since the last correct standard must be re-measured.

DISPOSAL

After test accuracy is confirmed by standard readings, empty contents of used test tubes slowly into toxic sink with running water. Rinse tubes and caps once and discard in trash.

PRECAUTIONS

TNT reagents contain cyanide compounds. These can be harmful if ingested, inhaled, or absorbed through the skin. Exercise caution when using these compounds. DO NOT allow these compounds to come in contact with any kind of acid, as toxic hydrogen cyanide gas might be generated.

CONVERSION OF TOTAL AMMONIA TO UNIONIZED AMMONIA

Total ammonia readings are calculated from an internal calibration on the Hach Spectrophotometer and converted to unionized ammonia using pH and the appropriate equilibrium coefficient. The following equation is used to determine what proportion of the total ammonia is unionized ammonia:

$$[NH_3] = [\text{total ammonia}] \times [1/(1+10^{(pK-pH)})]$$

Ammonia pK values:

	0 ‰	20‰	35‰
15°C	9.564	9.619	9.677
20°C	9.400	9.457	9.512
25°C	9.245	9.302	9.354

Example: Temp = 15°C. $[1/(1+10^{(9.677-8)})] = 0.021$

Total ammonia = 2 mg/l $0.021 \bullet 2 \text{ mg/l} = 0.041 \text{ mg/l } NH_3$

pH = 8